<u>PATENT</u> #Y2-0417-UNI Case #F7536(V)

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METHOD FOR PROCESSING OLIVES

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Sir:

Pursuant to rule 55(b) of the Rules of Practice in Patent Cases, Applicant(s) is/are submitting herewith a certified copy of the European Application No. 00303746.2 filed May 4, 2000, upon which the claim for priority under 35 U.S.C. § 119 was made in the United States.

It is respectfully requested that the priority document be made part of the file history.

Respectfully submitted,

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Bescheinigung

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Attestation

Die angehefteten Unterlagen stimmen mit der ursprünglich eingereichten Fassung der auf dem nächsten Blatt bezeichneten europäischen Patentanmeldung überein.

The attached documents are exact copies of the European patent application conformes à la version described on the following page, as originally filed.

Les documents fixés à cette attestation sont initialement déposée de la demande de brevet européen spécifiée à la page suivante.

Patentanmeldung Nr. Patent application No. Demande de brevet nº

00303746.2

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Blatt 2 der Bescheinigung Sheet 2 of the certificate Page 2 de l'attestation

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Anmelder: Applicant(s): Demandeur(s): UNILEVER PLC London EC4P 4BQ UNITED KINGDOM

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Method for processing olives

The present invention relates to a method for processing olives for obtaining an olive oil with an increased amount 5 of polyphenols.

Olive oil is a product which is of increasing interest, not only for its taste but also for the possible health improving properties. Some of these health improving 10 properties are attributed to the presence of so-called minor nutrients in the olive oil, in particular antioxidants such as polyphenols. It has recently been found that such anti-oxidants positively interfere with the body's cardiovascular system.

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- The present invention is directed towards increasing the amount of polyphenols in the olive oil. Polyphenols are known to be anti-oxidants which are present in olive fruits. Usually polyphenols are present not as a single compound but 20 as a mixture of different polyphenols. Polyphenols are compounds which share a phenolic hydroxyl group. Olive fruit originating polyphenols comprise oleuropein, aglycons,
- 25 The object of the present invention is to increase the level of polyphenols in olive oil, by means of a relative simple method. The object is further that such a simple method is applicable in conventional processes that are used for processing olives without any extra costs. Minimal
- 30 additional equipment is necessary.

tyrosol and hydroxytyrosol.



The conventional process for processing olives comprises the following steps:

- harvesting the olive fruits, removal of any leaves and washing of the olive fruits;
 - 2. crushing;
 - 3. malaxation;
 - 4. decanting the olive oil;
 - 5. centrifugation.

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According to the invention it has been found that the level of polyphenols in an olive oil can be increased by adding water to the olives during the crushing of the olives. It has been shown that the addition of water causes fine fruit

- 15 particles, which are hydrophilic, to be entrained in the added water, and thereafter in the produced olive oil. This effect will contribute to the polyphenols content of the olive oil.
- 20 Therefore, according to the invention a method is provided for processing olives obtaining an olive oil, comprising the steps of
 - a) crushing the olives through a sieve;
 - b) malaxation of the olive paste obtained in step a);
- 25 c) decanting the olive oil;

characterized in that

in step a) water is added in an amount of 1 to 40 % by weight based on the weight of the olives.

During step a) of the process olive oil fruits are crushed, preferably in a hammer mill equipped with a perforated sieve. The hammers rotate inside the sieve with a tip speed that is for example 65 m/sec. Conventionally the sieve will have a mesh of 6 to 8 mm. The present inventors have found that the polyphenols content can be further increased by using a sieve having a mesh of 1 to 6 mm, preferably 1 to 4 mm. By using a smaller sieve the fraction of smallest fruit particles will be maximized.

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The amount of water added is within the ranges indicated above. However, depending on the ripeness of the olive fruit, the amount of water added can be varied. The ripeness of the olive fruit is determined by means of the 15 ripeness index, as described for instance by Ranalli and Morelli, Leatherhead Food RA Food Industry Journal, Vol. 2, winter 1999.

The ripeness index defines the maturity of the olive as a 20 function of fruit color in both skin and pulp. Samples of olives, 100 for each variety, are taken at random and are classified into the following categories: 0 = olives with intense green or dark green epidermis (a); 1 = olives with yellow or yellow-green epidermis (b); 2 = olives with 25 reddish yellow epidermis (c); 3 = olives with reddish or light violet epidermis (d); 4 = olives with black epidermis, and totally white pulp (e); 5 = olives with black epidermis, and violet pulp to the midpoint (f); 6 = olives with black epidermis, and violet pulp almost to the 30 pit (g); 7 = olives with black epidermis, and totally dark

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pulp (h). The ripeness index is calculated from equation 1, where alphabetic variables indicate the number of fruits in each category.

5 RI =
$$(a \times 0 + b \times 1 + c \times 2 + d \times 3 + e \times 4 + f \times 5 + g \times 6 + h \times 7)/100$$
 [1]

Ripeness values are between 0 and 7.

10 It has been found that the higher the ripeness index, the less water will need to be added to the olives. Therefor, for olives having a ripeness index of 0 to 4, the water added is preferably 12 to 28 % by weight and for olives having a ripeness index of 4 to 7, the water added is preferably 1 to 12 % by weight.

It was further found that the crusher throughput is a factor that also influences the amount of polyphenols in the olive oil obtained. This optimum is influenced by the 20 type and maturity of the olives, as described above, and also by the choice of the mesh of the sieve.

According to a further aspect of the invention, the water added in step a) contains citric acid as a processing aid.

25 The citric acid will increase the amount of polyphenols in the final olive oil. A further advantage of the use of citric acid is that it has a protective function by chelating-metals, which prevents the metals of being catalysts for the oxidation of the polyphenols. The water

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preferably contains 0.5 to 7 % by weight citric acid, based on the weight of the water.

Preferably as the source of citric acid lemon juice is added. In general lemon juice contains about 7 % by weight citric acid. The lemon juice can be added as such to the water, as long as the citric acid content is within the ranges indicated above. It is also possible to add whole lemons or lemon parts to the water.

- In step b) malaxation of the olive paste obtained in step
 a) is carried out. Malaxation serves to favor the
 separation of the liquid phases from solids and to break up
 oil-water emulsions. It facilitates the coalescence of oil
 15 droplets and thereby directly influences the oil yield.
 Malaxation can proceed under conventional process
 conditions, for instance at 30 °C and 30 rpm for 30
 minutes.
- 20 In step c) olive oil is separated from solid phase and aqueous phase. In this step only the coarsest solid matter is removed. The fines, with a high polyphenols content, should be retained in the oil, preferably substantially remaining in the aqueous phase dispersed in the oil. This
- 25 step can be carried out by means of a centrifuge.

 Preferably, the decanting step is carried out such that the resulting olive oil has a residual water content of less than 3 wt.%, preferably 0.5 to 1 wt.%. When citric acid has been added to the water, the residual citric acid content
- 30 in the olive oil will be from 5 ppm to 5000 ppm.

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The polyphenols content can be optimized by means of the throughput through the decanter. In general a higher throughput will result in separation of only the heaviest 5 fraction and therefore more olive particles will remain in the olive oil.

After the decanting step an olive oil will be obtained which still contains some olive particles. The solids 10 content of the olive oil after decanting is preferably 0.05 to 5 wt.%, preferably 0.1 to 1 wt.%. The average particle size of the olive particles is less than 50 µm, preferably less than 20 µm, more preferably less than 5 µm. For some applications it can be desirable to have a clear oil. In 15 that case the olive oil can be purified further to remove at least part of the remaining olive particles, for instance by means of centrifugation. It has been found that even in that case an olive oil will be obtained that has a higher polyphenols content than those obtained with 20 conventional methods.

The present invention also relates to the olive oil obtained with the above described method. Generally, the olive oil of the invention has a polyphenols content higher 25 than 500 ppm. In case of the processing of unripe olives having a ripeness index of 0 to 4, the polyphenols content is higher than 500, preferably higher than 1000 ppm. In case of the processing of ripe olives having a ripeness index of 4 to 7, the polyphenols content is higher than 300

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ppm, preferably higher than 400 ppm. The oil obtained has a nice taste with minimal bitterness or astringency.

The total content of polyphenols in olive oils can be

5 established by standard methods, e.g. by the colorimetric
Gutfinger method as described in J.Am.Oil.Chem.Soc. 1981,

11, pp. 966-968, which method is based on the reaction of a
methanolic extract of olive oil and the Folin-Ciocalteau
reagent. This method has been used for establishing the

10 polyphenols values mentioned in the present patent
specification.

The olive oil of the invention can be used in the preparation of food products such as a spread, salad

15 dressing, mayonnaise or sauce. Spreads are food compositions which usually contain a substantial amount of fat, often 40 wt.% or more. Usually the fat consists of a liquid oil and a structuring fat which gives the fat blend a proper consistency. Sauces are meant to include any type of sauce,

20 for instance sauces that are ready to use, in particularly after having been heated, such as for instance tomato sauces. Processes for the manufacture of these products are well known in the art and need no illustration.

25 The olive oil can also be blended with vegetable oils, such as rapeseed oil, sunflower seed oil, soybean oil and corn oil. The invention is not limited to oils which are devoid of any polyphenols, either by nature or because of a refining process, but also of oils which contain polyphenols 30 of their own such as (extra) virgin olive oils. Examples of

other olive oils which can be mixed with the oil according to the present invention are an extra virgin olive oil, a fine virgin olive oil, a semi-fine or regular virgin olive oil, a refined olive oil or an olive residue oil but also an 5 olive oil blend, which contains part virgin olive oil and part refined olive oil.

According to a second embodiment of the present invention a method is provided comprising the steps of

- a) crushing olives through a sieve, wherein water is added in an amount of 1 to 40 % by weight, preferably 12 to 28 % by weight based on the weight of the olives;
 - b) malaxation of the olives;
- 15 c) separating olive kernel particles from the paste.

 The preferred crushing and malaxation conditions are the same as described above.

The olive paste obtained with this process has a high level 20 of polyphenols and is suitable for food applications, in particular in food products having a fat phase. Said paste can be used in food products, such as olive oils, spreads, mayonnaise, sauces, etc. to replace the fat phase normally used in the production of these products.

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Example 1

An industrial milling was applied, with a hammer crusher - equipped with a 4mm mesh sieve. 430 kg olives (Tsounati variety) were crushed in this mill while adding 70.5kg 30 water (16.4 wt.%). Malaxation of the resulting paste was

performed at 30 °C and 30 rpm for 30 minutes. The decanter was operated at such a speed that the residual water content of the oil was 3.2 wt.%. The obtained oil is characterized by the fact that it contains 3.2 wt.% water, 5 0.54 wt.% olive particles with an average particle size of 11.0 µm and 1100 ppm polyphenols. The taste of the product presented minimal bitterness and astringency.

Example 2

10 The oil from Example 1 is further processed as if a conventional process scheme was applied. The oil is centrifuged in a standard vertical centrifuge. The obtained reference oil is characterized by the fact that it contains only 0.28 wt.% water, 0.03 wt.% olive particles and 269 ppm 15 polyphenols.

Example 3

In the same mill mentioned in Example 1 414kg olives (Tsounati variety) were crushed while adding 118.4kg water 20 (28.6 wt.%). Malaxation of the resulting paste was performed at 30 °C and 30 rpm for 30 minutes. The decanter was operated at such a speed that the residual water content of the oil was 1.3 wt.%. The obtained oil is characterized by the fact that it contains 1.3 wt.% water, 25 0.32 wt.% olive particles with an average particle size of 13.6 µm and 868 ppm polyphenols. The taste of the product presented minimal bitterness and astringency.

Example 4

In a lab scale hammer mill, equipped with a 2 mm mesh sieve, 100 g olives (Koroneiki variety) were crushed while adding 30 g water (30 wt.%) containing 1.44 g citric acid (4.8 wt.%). Malaxation of the resulting paste was performed 5 at 30 °C for 30 minutes. After centrifugation of the paste, the obtained oil is characterized by the fact that it contains 960 ppm polyphenols and 2434 ppm citric acid. The presence of citric acid in the oil was not detected by tasting.



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Claims

- 1. Method for processing olives obtaining an olive oil, comprising the steps of
 - a) crushing the olives through a sieve;

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- malaxation of the olives obtained in step a); b)
- C) decanting the olive oil; characterized in that in step a) water is added in an amount of 1 to 40 % by weight, based on the weight of the olives.
- Method according to claim 1, wherein the olives are 2. crushed through a sieve having a mesh of 1 to 6 mm, preferably 1 to 4 mm.
- 3. Method according to claim 1 or 2, wherein the decanting step is carried out such that the resulting olive oil has a residual water content of less than 3 wt.%, preferably 0.5 to 1 wt.%.
- Method according to any of the preceding claims, which further includes removing at least part of the olive particles remaining in the olive oil.
- 5. Method according to any of claims 1 to 4, wherein olives having a ripeness index of 0 to 4 are processed, characterized in that water is added in an amount of 12 to 28 % by weight.

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- 6. Method according to any of claims 1 or 4, wherein olives having a ripeness index of 4 to 7 are processed, characterized in that water is added in an amount of 1 to 12 % by weight.
- 7. Method according to any of the preceding claims, wherein the water added in step a) contains citric acid, preferably in an amount of 0.5 to 7 % by weight, based on the weight of the water.
- 8. Method according to claim 7, wherein lemon juice is used as the source of the citric acid.
- 9. Olive oil obtainable with the method of claim 5, having a polyphenols content higher than 500 ppm, preferably higher than 1000 ppm.
- 10. Olive oil obtainable with the method of claim 6, having a polyphenols content higher than 300 ppm, preferably higher than 400 ppm.
- 11. Olive oil obtainable with the method according to any of claims 1 to 8, having a solids content after decanting of 0.05 to 5 wt.%, preferably 0.1-1 wt.%.
- Olive oil according to claim 11 containing olive particles having a particle size smaller than 50 μm,
 preferably smaller than 20 μm, more preferably smaller than 5 μm.

- 13. Food product containing an olive oil according to any or claims 9 to 12.
- 14. Food product according to claim 13, which is a vegetable oil, preferably an extra virgin olive oil.
- 15. Food product according to claim 13, which is a spread, mayonnaise, salad dressing or tomato sauce.
- 16. Method for preparing an olive paste, comprising the steps of
 - a) crushing olives through a sieve, wherein water is added in an amount of 1 to 40 % by weight, preferably 12 to 28 % by weight, based on the weight of the olives;
 - b) malaxation of the olives;
 - separating olive kernel particles from the paste.
- 17. Olive paste obtainable by the process of claim 16.
- 18. Food product containing an olive paste according to claim 17.



Abstract

It has been found that the level of polyphenols in an olive oil can be increased by adding water to the olives during the crushing of the olives. In particular the invention concerns a method for processing olives obtaining an olive oil, comprising the steps of

- a) crushing the olives through a sieve in the presence of water in an amount of 1 to 40 % by weight, based on the weight of the olives;
- b) malaxation of the olives obtained in step a);
- c) decanting the olive oil.